XVII. *On the Analyſis of a Mineral Subſtance from* New South Wales. *In a Letter from* Joſiah Wedgwood, *Eſq. F. R. S. and A. S. to Sir* Joſeph Banks, *Bart. P. R. S.*

Read April 15, 1790.

DEAR SIR, Etruria, March 12, 1790.

I HAVE the pleaſure of acquainting you, that the clay from Sydney-Cove, which you did me the honour of ſubmitting to my examination, is an excellent material for pottery, and may certainly be made the baſis of a valuable manufacture for our infant colony there. Of the ſpecies of ware which may be produced from it, you will have ſome idea from the medallions I have ſent for your inſpection.

The other mineral, which you favoured me with a ſpecimen of, ſeems to me to contain one ſubſtance hitherto unknown, and another not known in the ſtate of purity in which it is here found. I ſhall therefore take the liberty of ſtating, as conciſely as I can, the principal reſults of the experiments that have been made upon it by myſelf and my aſſiſtant Mr. CHISOLM; ſubmitting it to your judgement, whether any of them be of importance enough to be laid before the Royal Society.

I have the honour to be, &c.

JOS. WEDGWOOD.

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Analytical experiments on a Mineral from Sydney-Cove *in* New South Wales.

THIS mineral is a mixture of fine white ſand, a ſoft white earth, ſome colourleſs micaceous particles, and a few black ones reſembling black mica or black-lead; partly looſe or detached from one another, and partly cohering together in little friable lumps.

None of theſe ſubſtances ſeem to be at all acted upon by the nitrous acid, concentrated or diluted; nor by oil of vitriol diluted with about equal its meaſure of water; in the cold, or in a boiling heat; the mineral remained unaltered in its appearance, and the acids had extracted nothing from it that could be precipitated by alkali.

Oil of vitriol boiled upon the mineral to dryneſs, as in the proceſs of making alum from clay, produced no apparent change in it; but a lixivium made from this dry maſs with water, on being ſaturated with alkali, became ſomewhat turbid, and depoſited, exceeding ſlowly, a white earth in a gelatinous ſtate, too ſmall in quantity for any particular examination; but which, from its aſpect, from the manner in which it was obtained, and from the taſte of the lixivium before the addition of the alkali, was judged to be the aluminous earth.

The marine acid, during digeſtion, ſeemed to have as little action as the other two; but on pouring in ſome water, with a view only to dilute and waſh out the remaining part of the acid, a remarkable difference preſented itſelf; the liquor became inſtantly white as milk, with a fine white curdly ſubſtance intermixed; the ſtrong acid having extracted ſomething which the ſimple dilution with water precipitated.

The white matter being waſhed off, more ſpirit of ſalt was added to the remainder, and the digeſtion repeated, with a long tube inſerted into the mouth of the glaſs, ſo as nearly to prevent evaporation. The acid, when cold and ſettled fine, was poured off clear; and on diluting it with water, the ſame milky appearance was produced as at firſt.

The digeſtion was repeated ſeveral times ſucceſſively, with freſh quantities of the acid, till no milkineſs appeared on dilution. The quantity of mineral employed was 24 grains; and the reſiduum, after the operations, waſhed and dried, weighed ſomewhat more than 19 grains; ſo that about one-fifth of it had been diſſolved. In ſome parcels of the mineral, taken up promiſcuouſly, the proportion of ſoluble matter was much leſs, and in none greater. It is only the white part, and only a portion of this, that the acid appears to act upon: the white ſand, much of the white ſoft earth, and all the black particles, remain unaltered.

To try whether this tedious proceſs of ſolution could be expedited by triture or calcination, ſome of the mineral was rubbed in a mortar; and in doing this, it appeared pretty remarkable, that though the black part bore but an inconſiderable proportion to the reſt, yet the whiteneſs of the other was ſoon covered and ſuppreſſed by it, the whole becoming an uniformly black, ſhining, ſoft, unctuous maſs, like black-lead rubbed in the ſame manner; with a few gritty particles perceptible on preſſing hard with the peſtle. A penny-weight of this mixt, ſpread thin on the bottom of a porcelain veſſel, was calcined about an hour, with a fire between 30 and 40 degrees *; it became of an uniform, dull, white, or grey colour, excepting

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* By degrees of *fire*, or of heat *above ignition*, I mean thoſe of my thermometer;

excepting a very few, and very ſmall, ſparkling, black particles, ſuſpected to be thoſe which had eluded the action of the peſtle; it loſt in weight ſix grains, or one-fourth.

The mineral, thus ground and calcined, was found to be juſt as difficult of ſolution as in its crude ſtate; with this additional diſadvantage, that the undiſſolved fine particles are indiſpoſed to ſettle from the liquor.

In all the experiments of diſſolution, as often as the heat was at or near the boiling point of the acid, frequent, and pretty ſingular, burſts or exploſions happened, though the matter lay very thin in a broad-bottomed glaſs. They were ſometimes ſo conſiderable as to throw off a porcelain cup with which the glaſs was covered, and once to ſhatter the glaſs in pieces. In a heat a little below this, the extraction ſeemed to be equally complete, though more ſlow; but a heat a little below that in which wax melts, or below 140° of FAHRENHEIT's thermometer, appeared inſufficient.

To determine the degree of dilution neceſſary for the precipitation of the diſſolved ſubſtance, and whether the precipitation by water be total, a meaſure of the ſolution was poured into a large glaſs, and the ſame meaſure of water added repeatedly. The third addition of water occaſioned a ſlight milkineſs, which increaſed more and more to the ſixth. The liquor being then filtered off, another meaſure of water produced a little freſh milkineſs; and an eighth rather increaſed it; a ninth and a tenth had no effect. The liquor being now again paſſed through a filter, ſolution of ſalt of tartar did not in the leaſt alter its tranſparency; ſo that, after the ſolution

meter; and ſome idea may be formed of their value, by recollecting, that they commence at viſible redneſs; and that the extreme heat of a good air-furnace, of the common conſtruction, is 160°, or a little more.

has been diluted with eight or nine times its meaſure of water, there is nothing left in it that alkali can precipitate.

From the manner in which the ſolution is neceſſarily prepared, it cannot but contain a great redundance of acid; for the ſmall quantity of acid, ſufficient for holding the ſoluble part ſuſpended, would be ſoaked up or entangled by the undiſſolved part, ſo as ſcarcely to admit of any being poured off; and it cannot be diluted, or waſhed out, but by the ſtrong acid itſelf. The ſolution with which the above experiment was made was reckoned to have only about ſix grains of the ſoluble matter to three ounces of ſpirit of ſalt, having been prepared by digeſting that quantity of the ſpirit by half an ounce at a time on thirty grains of the crude mineral.

A *ſaturated* ſolution was obtained by digeſting, in a ſmall portion of the ſolutions thus prepared, the precipitate thrown down by water from the larger portions till the acid would take up no more. A ſolution, thus ſaturated, cannot bear the ſmalleſt quantity of water, a ſingle drop, on the firſt contact, producing a milky circle round it.

Examination of the above ſubſtance, extracted from the mineral by marine acid, and precipitated by water.

This ſubſtance, waſhed and dried, is indiſſoluble in water, as indeed might be expected from the manner of its preparation.

Nor is it acted upon by the nitrous or vitriolic acids, concentrated or diluted, cold or hot; nor by alkaline ſolutions, mild or cauſtic, of the volatile or fixed kind.

It is diſſolved by ſtrong marine acid, but not without the aſſiſtance of nearly the ſame degree of heat that is neceſſary for its extraction from the mineral. From this ſolution it is precipitated by water; and, after repeated diſſolutions and precipitations, it appears to have ſuffered no decompoſition or change.

Spirit of nitre, added to the ſaturated ſolution, makes no precipitation; and if the quantity of nitrous acid exceeds, or at leaſt does not fall much ſhort of, that of marine acid in the ſolution, the mixture ſuffers no precipitation from water. Nor does any precipitation happen, though the nitrous ſpirit be *previouſly* mixed with even a large quantity of water; provided the quantity of ſolution added to it does not exceed that of the nitrous ſpirit in the mixture. The appropriate menſtruum for this ſubſtance (that is, for keeping it in a ſtate of dilute ſolution) appears therefore to be aqua regia; and the due proportions of the two acids, of any given ſtrength, might be determined, if neceſſary, with greater accuracy and facility for this than for any other body I know of; becauſe, if there be even a very minute ſurplus of marine acid in the ſolution, that ſurplus will inſtantly betray itſelf on dropping a little into water, all that was diſſolved by *it*, and no more, being precipitated by the water. It may be obſerved, however, that where an addition of nitrous acid is uſed, a *ſaturated* ſolution cannot be obtained (unleſs by ſubſequent evaporation), the ſame quantity of marine acid being neceſſary with as without that addition: the change, or modification, which the nitrous acid produces in the marine, ſerves, in the preſent inſtance, not for effecting the ſolution, as in the caſe of gold and ſome other metals, but merely for enabling it to bear water without depoſiting its contents.

Oil of vitriol, dropped into the ſaturated marine ſolution, occaſions no change till its quantity comes to be about equal to that of the ſolution; a conſiderable effervefcence and heat are them then produced, the liquor becomes milky, and the marine acid is extricated in its uſual white fumes. The mixture, heated nearly to boiling, becomes tranſparent, and afterwards continues ſo in the cold. This vitriolic ſolution is precipitated by water, and the precipitate is re-diſſolved by marine acid.

The ſaturated marine ſolution is indiſpoſed to cryſtallize. By continued evaporation in gentle heat, it becomes thick and butyraceous, and in this ſtate it ſoon liquefies again on expoſure to the air. The butyraceous maſs, in colour whitiſh or pale yellow, is not corroſive, like the ſimilar preparations made from ſome metallic bodies; nor is it more pungent in taſte, but rather leſs ſo than the combination of the ſame acid with calcareous earth. In a heat increaſed nearly to ignition, the acid is diſengaged, and riſes in white fumes, which, received in a cold phial, condenſe into colourleſs drops, without any appearance of ſublimate. From the remaining white maſs, ſpirit of nitre extracts ſo little as to exhibit only a ſlight milkineſs on adding alkali; a proof that nearly all the marine acid had been expelled; for, while that acid remains, the whole is diſſoluble by the nitrous.

The ſubſtance in queſtion is not precipitated by Pruſſian lixivium. A drop or two of the lixivium do indeed occaſion a little white or bluiſh-white precipitation in the *ſaturated* marine ſolution; but in the more dilute no turbidneſs appears till the quantity of lixivium is ſuch as to produce that effect by its mere water; and when the precipitate has at length been formed, it re-diſſolves in marine acid as eaſily as that made by water; whereas the precipitates reſulting from the union of the Pruſſian matter are not acted upon by acids, till that matter has been extracted from them by an alkali. For further ſatisfaction in this important point, the experiment was repeated with a ſolution in aqua regia. Here the Pruſſian lixivium, in whatever quantity it was added, occaſioned no precipitation at all (only the uſual bluiſhneſs, ariſing from the iron always found in the common acids); and pure alkali, added afterwards, precipitated the original white ſubſtance unchanged.

The following experiments of precipitation by alkalies were made with the marine ſolution, before the effect of an addition of nitrous acid had been diſcovered; and they were made with ſo much care and attention, that it was not thought neceſſary to repeat them afterwards. To obviate, as much as poſſible, the equivocal reſults that might ariſe from *water* contained in the precipitants, the different alkalies were applied in the dryeſt ſtate I could reduce them to; *viz.* pure *ſalt of tartar*, kept for ſome time in a heat juſt below redneſs; cryſtals of *marine alkali*, melted and dried in the ſame manner; *volatile alkali* in cryſtals, a little ſurplus acid being, in this inſtance, previouſly added to the ſolution, to counteract the water of cryſtallization in the alkali; *ſalt of tartar cauſticated* by quicklime, and haſtily evaporated to dryneſs; the *marine alkali cauſticated* in like manner; and the vapour of *cauſtic volatile alkali* ariſing, with a very gentle heat, from a retort into a phial containing the ſolution. All theſe alkalies occaſioned copious precipitations. All the precipitates, after waſhing and drying, were found to re-diſſolve in marine acid; and, from all theſe ſolutions, the original ſubſtance was precipitated, unaltered, on diluting them with water.

In ſtrong fire, from 142 to 156 degrees, this ſubſtance diſcovers a much greater fuſibility than any of the known ſimple earths. In a ſmall veſſel, made of tobacco-pipe clay, it melted, and glazed the bottom; and on a bed of powdered flint, preſſed ſmooth in the manner of a cupel, it did the ſame. Magneſia, or chalk, would indeed vitrify in the clay veſſel; but on flint, no one of the known earths ſhews any tendency to vitrification in that heat *. In a cavity, ſcooped in a lump of chalk, this ſubſtance, in the heat above mentioned, run into a ſmall round bead, ſmooth, whitiſh, and opaque, not in the leaſt adhering to the calcareous maſs. On a bed of powdered quicklime it formed a browniſh ſcoria, which in great part had ſunk into the lime, and ſeemed to have united with it. On Mr. HENRY's magneſia, uncalcined, it melted and ſunk in completely, leaving only a ſlight browniſh ſtain on the ſurface where it had lain. On beds of the baroſelenite and barytic quicklime, it likewiſe melted and ſunk in, leaving a diſcoloured ſpot behind; but whether it really united with the ſubſtrata, or only penetrated into their interſtices, could not be determined with certainty, on account of the ſmallneſs of the quantity of the mineral I had to work upon.

On a bed of powdered charcoal, in a crucible cloſely luted, this ſubſtance likewiſe melted; and therefore it may be pre-

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* It may be proper juſt to mention, that I find this to be a very commodious and ſure method of trying, in ſmall, whether any given earthy body be fuſible with other earths. If the body is diſpoſed to vitrify with any proportion of clay or flint, for inſtance, it will equally vitrify when a little of it is applied, or even duſted only, on the bottom of a ſmall cup made of clay, or on a ſmooth cloſe bed of finely powdered flint. The body, in this mode of application, ſeems to unite with only juſt ſo much of the matter of the ſubſtratum as is requiſite for their moſt perfect fuſion together, and has nothing elſe in contact with it, ſo that no deception can ariſe; whereas, if *mixed* with the ſame matter, there might be no appearance of fuſion, unleſs certain favourable proportions of the two ſhould chance to be hit upon; and even then, if the quantity be ſmall, it would not be certain but that the fuſion might have originated from the matter of the crucible.

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ſumed not to have owed its fuſion, in the above experiments, to the ſame cauſe to which ſome of the common ſimple earths, in certain circumſtances, owe theirs, namely, their union with the matter of the veſſel or ſupport, that is, with an earth or earths of a different kind from themſelves; but to poſſeſs a fuſibility ſtrictly its own, which takes place in a fire of 150 degrees, or perhaps leſs.

As charcoal in fine powder aſſumes a kind of fluidity in the fire, ſimilar to that which powdered gypſum exhibits in a ſmall heat, its ſurface had changed from concave to horizontal, and the bead had ſunk to the bottom; it was rough and black on the outſide, and whitiſh within. On repeating the experiment in a cavity ſcooped in a piece of charcoal, the reſult was a blackiſh bead like the former, only ſmooth on the outſide, with ſomething of metallic brightneſs, not unlike that of black-lead. Both beads were very light, and had a conſiderable cavity within. All the internal part was whitiſh, without the leaſt metallic aſpect; and the external gloſſy blackneſs appeared to be only the ſtain which charcoal powder communicates, in ſtrong fire, to ſome earthy bodies that have a tendency to vitrify. By boiling in concentrated marine acid a part of the beads was diſſolved, precipitable as at firſt by water; but an accident prevented the proceſs from being continued ſufficiently to determine whether the whole could be diſſolved or not.

By this fuſibility in the fire; ſolubility in one only of the common mineral acids, and parting with the acid in a heat below ignition; precipitability by water, and non-precipitability by Pruſſian lixivium; this ſubſtance is ſtrongly diſcriminated from all the known earths and metallic calces. And as it ſuffers no decompoſition from any of the alkalies, in any of the uſual modes of application, I preſume, it cannot be conſidered as a combination of any of thoſe earths or calces with any of the known acids; for all the combinations of this kind would, in one or other of the above methods of trial, have had the earth or metal diſengaged from the acid.

Whether this ſubſtance belongs to the *earthy* or *metallic* claſs, I cannot abſolutely determine; but am inclined to refer it to the earthy; becauſe, though brought into perfect fuſion, in contact with inflammable matter, and in cloſe veſſels, it does not aſſume the appearance which metallic bodies do in that circumſtance.

Examination of the black particles.

Theſe particles, which bore but a very ſmall proportion to the other matter, were in form of ſhining black ſcales, very thin, and very light. One grain weight of them, carefully picked out, expoſed to a fire which was gradually raiſed to about 90°, and continued in all about 40 hours, in a veſſel looſely covered, was almoſt wholly diſſipated, and what little remained was perfectly white. Marine acid had no effect on it.

Fifteen grains of the entire mineral loſt, in the ſame fire, three grains. After ſeparating from another portion of the mineral, by waſhing and otherwiſe, a conſiderable quantity of the white matter, 15 grains of the remainder, containing of courſe more than its due proportion of the black, loſt five grains; ſo that it ſeems principally to be the ſubſtance on which the blackneſs depends that is deſtroyed or diſſipated by fire. The ſame quantity, 15 grains, of common black-lead loſt in the ſame fire above 14 grains, the reſiduum weighing leſs than one grain.

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Though no concluſion can be drawn from theſe experiments reſpecting the comparative *loſs* of black-lead and the *pure* black matter of this mineral, on account of the heterogeneous parts intermixed with the latter, the *colour* of the reſidua ſeems to afford a ſufficient diſcrimination between them; that of black-lead being dark reddiſh brown, but the others purely and uniformly white.

As this ſubſtance could not now be ſuppoſed to be either iron mica, or the common kind of black-lead, ſuſpicion fell upon molybdæna. I had not, at that time, had an opportunity of procuring a ſpecimen of molybdæna to compare it with; but from the ſingular and ſtrongly-marked properties of the molybdænic acid, diſcovered by SCHEELE, it was judged, that a very ſmall quantity of it, when diſengaged from the ſulphur with which it is naturally combined, would eaſily be diſtinguiſhable.

HJELM's proceſs for diſengaging the ſulphur, by repeatedly burning linſeed oil upon the molybdæna in a crucible, and afterwards abſtracting ſucceſſive quantities of the ſame oil from it in a retort, was tried on a portion of the Sydney-Cove mineral, from which much of the white matter had been ſeparated as above mentioned. The black coal, remaining in the retort, became yellow by calcination, as that of molybdæna ſhould do; but in this yellow powder, no veſtige of molybdænic acid could be diſcovered.

Another quantity of the mineral was ſubmitted to SCHEELE's own proceſs, *viz.* repeated abſtractions of *diluted* nitrous acid; but, inſtead of becoming whiter every time, and at length white as chalk, which molybdæna ſhould do, the blackneſs of this matter continued unaltered to the laſt.

There is one circumſtance in Mr. Scheele's experiments, which, though omitted by thoſe who have given abſtracts of them, may deſerve, on the preſent occaſion, to be more particularly noticed. He reduced the molybdæna into fine powder, and poured upon it *concentrated* nitrous acid: "the mixture," he ſays, "was hardly lukewarm in the retort, when it paſſed all "together into the recipient with great heat;" and it was for *this* reaſon that he afterwards uſed *diluted* acid. Preſuming that this violent action of the concentrated nitrous acid might afford a deciſive criterion of molybdæna, I had the black reſiduum, after five or ſix abſtractions of the diluted acid, ground fine upon a levigating glaſs, and returned into the retort, with ſix times its weight of ſmoking ſpirit of nitre. The heat was increaſed cautiouſly far beyond lukewarm, but no commotion could be perceived, except the exploſions already mentioned, which always took place when the mixture was near boiling. The diſtillation was continued to dryneſs, and repeated five times with the ſmoking acid; but the mineral remained juſt as black as it was at firſt.

Now, as Scheele's molybdæna is ſlowly decompoſed by the diluted nitrous acid, and rapidly acted upon by the concentrated acid, while the black part of this mineral obſtinately reſiſts both, I think we cannot heſitate to conclude, that this black ſubſtance is not Scheele's molybdæna. There are ſome other circumſtances which confirm this concluſion, although, taken ſingly, they would not, perhaps, be of much weight, conſidering the great proportion of other matter here mixed with the black. The principal of theſe circumſtances are, that it yields no flowers before a blow-pipe, and that its particles ſeem to have no flexibility or elaſticity, the only difficulty

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of reducing it into fine powder ariſing from a property of another kind, unctuoſity.

The difference, above taken notice of, between this black matter and common black-lead, conſiſts only in the former leaving on calcination a white ſubſtance, ſeemingly ſiliceous, and the latter a brown ferrugineous one. In their aſpect, unctuoſity, reſiſtance to acids, and the volatility (in open fire) of that part in which the blackneſs conſiſts, they perfectly agree; and they appear to agree alſo in the nature or conſtitution of this volatile part; for the Sydney-Cove mineral, as well as black-lead, deflagrates and effervеſces very ſtrongly with nitre, produces an hepatic impregnation on fuſion with vitriolated alkali, but none with pure alkali, and is manifeſtly rich in inflammable matter, without ſulphur.

It ſeems, therefore, that this ſubſtance is a *pure* ſpecies of plumbago, or black-lead, not taken notice of by any writer I have met with. FOURCROY, in the laſt edition of his Chemiſtry, looks upon iron as an eſſential component part of black-lead, to which, accordingly, he gives a new name expreſſive of that metal, *carbure de fer*. LAVOISIER, in his Elements of Chemiſtry, lately publiſhed, mentions a *carbure* of zinc alſo, and ſays that both theſe carbures are called plumbago, or black-lead. The quantity of mineral I had been furniſhed with was too far exhauſted, before I met with this obſervation, to admit of any further experiments, for determining the preſence of zinc in it; but thoſe already ſtated, with the recollection of ſome circumſtances attending them, perſuade me, that *that* metallic body has no ſhare in its compoſition. Neither before the blow-pipe, nor in calcination, was there any appearance of the peculiar flame, or flowers, by which

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zinc is ſo ſtrongly characteriſed: if any ſuch appearance had taken place, it could not have eſcaped notice, as ſome of the calcinations were particularly attended to during the proceſs, though with a different view, the diſcovery of ſulphur or arſenic. The white matter which remains after the calcination is certainly not calx of zinc, for it was not acted on by ſpirit of ſalt, cold or hot, while the calces of zinc are diſſolved rapidly by that acid, even in the cold.